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Indian Standard

METHODS OF TESTS FOR
CHROMATE CONVERSION COATINGS ON
ZINC AND CADMIUM SURFACES

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METHODS OF TESTS FOR CHROMATE CONVERSION COATINGS ON ZINC AND CADMIUM SURFACES

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Indian Standard

METHODS OF TESTS FOR CHROMATE CONVERSION COATINGS ON ZINC AND CADMIUM SURFACES

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 October 1977, after the draft finalized by the Metallic Finishes Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 The methods of testing chromate conversion coatings relate to evaluation of the protective value of the chemical and electrochemical conversion coatings produced by chromate treatments of zinc and cadmium surfaces. Passivation has little effect on coating thickness of zinc and cadmium. The protective value is usually determined by humidity or salt spray test. Besides, it is important to know the presence of the colourless chromate coating and hexavalent chromium in both colourless and coloured chromate coatings. The adhesion test has also been included as the chromate coatings should be adherent and possess adequate abrasion resistance.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard covers the methods for checking the required characteristics for chemical conversion coatings obtained by chromate treatments on zinc and cadmium surfaces.

2. APPLICABILITY

2.1 The methods covered in this standard are applicable to chromate coatings of the colourless (both one and two-dip) iridescent yellow or bronze, olive drab, black, colourless anodic, yellow or black anodic types, and of the dyed variety, when applied to surfaces of electro-deposited zinc, hot dipped zinc, electro-deposited cadmium and zinc die castings.

*Rules for rounding off numerical values (*revised*).

2.2 In order to avoid risk of uncertainties in results, these tests shall be carried out within the following time limits:

- a) Minimum 24 hours after the application of the chromate conversion coatings for all tests (3 to 9),
- b) Maximum 3 days for tests specified in 3 and 4, and
- c) Maximum 30 days for tests specified in 4, 6 and 7.

3. TEST FOR PRESENCE OF A COLOURLESS CHROMATE COATING

3.1 Test Solution — Dissolve 50 g of lead acetate trihydrate [$\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$] in 1 litre of distilled or deionized water. The pH of the solution shall be between 5.5 and 6.8.

3.1.1 Any white precipitate formed during the initial preparation of the solution may be dissolved by small additions of acetic acid, provided that the pH is not reduced to a value below 5.5. Upon formation of a white precipitate thereafter, the solution shall be discarded.

3.2 Procedure

3.2.1 Place 1 drop of the test solution on the surface to be tested. In the case of a zinc substrate, observe the spot for 3 minutes. The presence of a colourless chromate conversion coating will be indicated when the formation of a dark or black stain is delayed by at least 1 minute after application of the test solution. Delay of black stain formation by more than 3 minutes may be indicative of supplementary coatings, such as wax or oil.

3.2.2 In the case of a cadmium substrate, observe the spot for 1 minute. The presence of a colourless chromate conversion coating will be indicated when the formation of a dark or black stain is delayed by at least 6 seconds. Delay of black stain formation by more than 1 minute may be indicative of supplementary coatings, such as wax or oil.

3.2.3 Zinc and cadmium surfaces which have not been treated with a chromate conversion coating will react with the lead acetate solution to form a black stain within 2 to 5 seconds.

3.2.4 No significance should be placed upon any differences in reaction time to black spot formation between 1 and 3 minutes on a zinc substrate with colourless chromate conversion treatment, or between 6 and 60 seconds on a cadmium substrate with colourless chromate conversion treatment. Factors, such as substrate surface texture (roughness), chromate film thickness variations (due to processing conditions), variations in the ambient temperature of the test, and precise pH control of the lead acetate solution have an influence on the reaction time, and

therefore preclude any rating based upon times beyond those already stated. Similarly, the test may not be used to compare various types of chromate treatment solutions.

NOTE — Both colourless and coloured chromate conversion coatings which have lost their protective value through dehydration from exposure to temperatures in excess of 60°C will form black stain in less than 60 seconds on zinc, and less than 5 seconds on cadmium.

4. TEST FOR PRESENCE OF HEXAVALENT CHROMIUM IN BOTH COLOURLESS AND COLOURED CHROMATE COATINGS

4.1 Test Solution — Dissolve 0.4 g of diphenylcarbazine in a mixture of 20 ml of acetone and 20 ml of ethanol. After dissolution, add 20 ml of 75 percent phosphoric acid and 20 ml of distilled water. The solution shall be prepared not more than 8 h prior to use.

4.2 Procedure

4.2.1 Place 1 to 5 drops of the test solution on the surface to be tested. If hexavalent chromium is present, a red to violet colour will appear within a few minutes. Any colour which appears much later, for example, on drying, may be disregarded.

4.2.2 For comparative purposes, a surface that is known not to have been chromate treated may be similarly tested.

5. DETERMINATION OF HEXAVALENT CHROMIUM CONTENT OF COLOURED CHROMATE COATINGS — DIPHENYLCARBAZIDE (COLORIMETRIC) METHOD

5.1 Reagents.

5.1.0 During analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

5.1.1 Dilute Sulphuric Acid — 1 : 3 (v/v).

5.1.2 Diphenylcarbazine Solution — Dissolve 0.50 g of diphenylcarbazine in 50 ml of acetone; dilute slowly, while stirring, with 50 ml of water (rapid mixing may result in precipitation of diphenylcarbazine). For maximum stability, keep under refrigeration in an amber-coloured glass bottle.

5.1.3 Phosphate Buffer Solution — Dissolve 55 g of sodium dihydrogen orthophosphate monohydrate ($\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$) in 100 ml of water.

5.1.4 Standard Potassium Dichromate Solution — Dilute 2 ml of standard potassium dichromate solution (0.1 N) to 1 litre. One millilitre of this solution contains 3.47/μg of chromium. A solution containing 9.90 mg/l of potassium dichromate contains 3.5 μg/ml of chromium.

5.2 Apparatus

5.2.1 Ordinary laboratory apparatus and photoelectric colorimeter or spectrophotometer may be used. With a photoelectric colorimeter, use a filter having a mean transmission of 520 nm. A wavelength of 540 nm shall be set for a spectrophotometer. An absorption cell with an optical path length of 1 cm shall be used in either case.

5.3 Procedure for Calibration

5.3.1 *Preparation of Calibration Curve* — To a series of five 250-ml volumetric flasks, add respectively 0, 10, 20, 30 and 40 ml of the standard potassium dichromate solution. Add to each 100 ml of water and 3 ml of dilute sulphuric acid. Take each flask through the following steps, one flask at a time, add 3 ml of the diphenylcarbazide solution and mix well. Exactly 2 minutes after mixing, add 25 ml of the phosphate buffer solution, dilute to the mark and mix.

5.3.2 Transfer a portion of each solution to an absorption cell and read in the colorimeter or spectrophotometer. Each reading shall be completed within 25 minutes after addition of the diphenylcarbazide solution because the colour is not stable. Subtract the blank (0 ml) from each reading and plot the differences against micrograms of chromium per 250 ml.

5.4 Blank Test — Carry out a blank test on the reagents simultaneously with the determination.

5.5 Procedure for Testing — Prepare a test piece measuring approximately 50×100 mm. Heat 50 ml of distilled water to boiling in a 400-ml beaker, then introduce the test piece. Leach for exactly 5 minutes while the water continues to boil, remove the test piece, rinse into the beaker, cool the beaker and contents to room temperature, add 3 ml of dilute sulphuric acid, mix, transfer to a 100-ml volumetric flask, add 3 ml of diphenylcarbazide solution and mix well. Exactly 2 minutes after mixing, add 25 ml of phosphate buffer solution, dilute to the mark and mix. Two minutes after the addition of the diphenylcarbazide solution, transfer a portion of the solution to an absorption cell and read in the colorimeter or spectrophotometer. Convert the reading, using the appropriate calibration curve, into micrograms per 100 ml.

5.6 Calculation — Deduct the reading obtained for the blank and determine the hexavalent chromium content of the film by reference to the calibration curve or by the use of a factor derived from it. Divide the hexavalent chromium content by the surface area of the test piece to obtain the hexavalent chromium content in milligrams per square centimetre.

6. DETERMINATION OF TOTAL CHROMIUM CONTENT OF COLOURED CHROMATE COATINGS-DIPHENYLCARBAZIDE (COLORIMETRIC) METHOD

6.1 Reagents

6.1.0 During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

6.1.1 *Standard Potassium Dichromate Solution* — see 5.1.4.

6.1.2 *Dilute Sulphuric Acid* — 1:3 (v/v).

6.1.3 *Phosphate Buffer Solution* — see 5.1.3.

6.1.4 *Diphenylcarbazide Solution* — see 5.1.2.

6.1.5 *Ammonium Persulphate* [(NH₄)₂S₂O₈] — solid.

6.1.6 *Silver Nitrate Solution* — (0.1 N) 17 g/l.

6.1.7 *Sodium Hydroxide Solution* — (6 N) 240 g/l.

6.2 Apparatus — see 5.2.

6.3 Procedure for Calibration

6.3.1 *Preparation of Calibration Curve* — To a series of five 250-ml volumetric flasks, add respectively 0, 10, 20, 30 and 40 ml of the standard potassium dichromate solution. Add to each 100 ml of water and 3 ml of dilute sulphuric acid. Take each flask through the following steps, one flask at a time; add 3 ml of the diphenylcarbazide solution and mix well. Exactly 2 minutes after mixing, add 25 ml of the phosphate buffer solution, dilute to the mark and mix.

6.3.2 Transfer a portion of each solution to an absorption cell and read in the colorimeter or spectrophotometer. Each reading shall be completed within 25 minutes after addition of the diphenylcarbazide solution because the colour is not stable. Subtract the blank (0 ml) from each reading and plot the differences against micrograms of chromium per 250 ml.

6.3.3 *Blank Test* — Carry out a blank test on the reagents simultaneously with the determination.

6.4 Procedure for Testing — Prepare a test piece measuring approximately 50 × 100 mm. Heat 25 ml of dilute sulphuric acid to 40 to 50°C and pour into a beaker containing the test piece. Swirl continuously so that all surfaces are exposed to acid for 10 to 15 seconds. Remove the test piece, and rinse into the beaker. Dilute to about 175 ml, and add about 0.1 g of the ammonium persulphate and 3 ml of the silver nitrate solution. Then add boiling chips and boil for 30 minutes. Cool, and adjust the pH to 1.5 to 1.8 with the sodium hydroxide solution (about 20 ml; to measure the pH, transfer a drop to pH paper — do not immerse the paper in the solution). Transfer to a 250-ml volumetric flask. Rinse the beaker twice into the flask with 10 to 15 ml of water, add 3 ml of

diphenylcarbazine solution, and mix. Exactly 2 minutes after addition of the diphenylcarbazine solution, add 25 ml of phosphate buffer solution, dilute to the mark and mix. Transfer a portion of the solution to an absorption cell and read in the colorimeter or spectrophotometer. Convert the reading, using the appropriate calibration curve, into micrograms per 250 ml.

6.5 Calculation — Deduct the reading obtained for the blank and determine the total chromium content of the film by reference to the calibration curve or by use of a factor derived from it. Divide the total chromium content by the surface area of the test piece to obtain the total chromium content in milligrams per square centimetre.

7. DETERMINATION OF COATING MASS PER UNIT AREA OF BOTH COLOURLESS AND COLOURED CHROMATE COATINGS

7.1 This shall be done in accordance with 'Indian Standard methods of testing conversion coating mass per unit area by gravimetric method (*under preparation*)'.

NOTE — Until this standard is published, the method of testing conversion coatings shall be as agreed to between the purchaser and the supplier.

8. ADHESION TEST

8.1 Test adhesion of a coloured chromate coating by rubbing the surface with a soft white tissue paper or with a gritless gum eraser, using normal hand pressure (about 10 strokes). There shall be no appreciable staining on the tissue paper when this is used for the test, nor shall the chromate coating be removed or worn through to the underlying zinc or cadmium when rubbed with the paper or the eraser.

9. HUMIDITY OR SALT SPRAY TEST

9.1 Humidity Test

9.1.1 Apparatus — A heat insulated chamber:

- a) A fan to circulate the air in the chamber,
- b) A non-metallic and non-corrosive support for the specimen near the centre of the chamber.

9.1.2 Temperature of Test — The test shall be conducted at a temperature of $55 \pm 2^\circ\text{C}$, followed by cooling to 30°C .

9.1.3 Humidity — The relative humidity shall not be less than 95 percent.

9.1.4 Test Cycle — The coating shall be subjected to the above-mentioned temperature and humidity conditions for 16 hours continuously. The source of heat shall then be turned off. Circulation of the air shall be maintained. The temperature shall be allowed to fall to 30°C . The article shall be kept at this temperature for five hours. The coating shall be examined after each cycle. Appearance of dark brown film after two cycles constitutes failure.